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Double cantilever beam tests on a viscoelastic adhesive: effects of the loading rate

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Abstract

Adhesive bonding technology is a promising alternative to traditional joining techniques. Indeed, bonded joint shows higher strength and fatigue life than bolted or riveted joints having identical weight. However, bonded joints are sometime reputed to be little reliable since significant dispersion could be observed while measuring their strength but also due to strong sensitivity to adhesion defect and poor surface preparation. Damage tolerance philosophy is now recommended for more reliable design of critical bonded parts by precise prediction of decohesion initiation and propagation along the bondline.
Double cantilever beam (DCB) test is the most popular method to characterize the decohesion resistance of bonded interface by measuring their fracture energy or their R-curve in case significant nonlinear behaviour is observed. These past years, several efficient analysis techniques have been proposed to evaluate the fracture energy but also some optimization techniques to identify more complex interface behaviour. However, most of these techniques consider non time dependent behaviour while thermoset adhesives are known to be viscoelastic and in some condition can also show viscoplastic behaviour. Such effects are important to evaluate when bonded joint sustain stationary loads since they could lead to delayed fracture and slow crack growth.
In the present work, we evidence some strain rate sensitivity at the bondline scale by performing DCB test under different opening rate conditions. At first, the viscoelastic behaviour of the adhesive is studied by performing creep test in a Dynamic Mechanical Analyser. The DCB tests results are interpreted with several methods including the Simple Beam Theory. It is shown that fracture energy is not an appropriate quantity to evaluate the crack propagation condition.

Keywords: Viscoelastic behaviour; Double Cantilever Beam

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Introduction

Double Cantilever Beam (DCB) test is the most common test used to determine the fracture toughness of adhesive bonds since the 1960s and the work of Ripling et Mostovoy (1964). Its testing procedure has been approve by a large majority due to its easily manufactured specimens and a simple standardised analysis (ASTM D3433) as can be seen in the work of Blackman and Kinloch (2000 and 2001) and Salem et al. (2013 and 2014). The sample consists of two flexible adherends bonded along part of their length. During Double Cantilever Beam test a force is applied normally to the bond surface at one end of each adherend. The fracture toughness of the specimen Gc can then be calculated using Simple Beam Theory (SBT) after evaluation of the joint's compliance. However this theory, though used on most on the employed adhesive, is restricted to those exhibiting linear elastic behavior (Irwin and Kies, 1952). Indeed in the case of viscoelastic adhesive, such as the own studied in this paper, time dependent damage will laid to a delayed fracture nucleation, mechanism very different from the one described in SBT.

Due to its large range of use, many studies have been performed on DCB tests over the year, still the effect of loading rate on the behaviour of the bond remains a seldom investigated field. Needleman and Rosakis (1999) have shown that loading rate has an unneglectable effect on crack initiation and the maximum stress supported by the bond and a few papers have shown inside on the influence of a high loading rate on the fracture of the joint, the first effect being a non-stable crack growth (Blackman et al.,1995, 2009, 2011).Those studies took an interest on the variation of Gc with loading rate, evidencing a drastic loss of fracture energy at high strain rate.

1. Experimental aspects

The experimental part of this study is divided into two parts: characterization of the bulk adhesive through DMA (Dynamic Mechanical Analysis) and creep tests and study of the crack propagation in adhesive joints through DCB (Double Cantilever Beam) tests.

1.1. Bulk adhesive and creep tests

The SW2216 – 3M© adhesive studied here is a two components epoxy paste used in aerospace applications. Resin and hardener are mixed and deposited on the adherends following supplier recommendations by using a SEMCO® 250-A pneumatic gun. Crosslinking is effected at room temperature (ca. 23°C) during five days, then a postcure is performed by heating the specimen during an hour at 66°C. Two millimeters thick dogbone samples (ISO 527-2/B) were obtained by applying pressure (2 bar) during crosslinking to the resin placed in a PTFE mold lying between two aluminum plates.

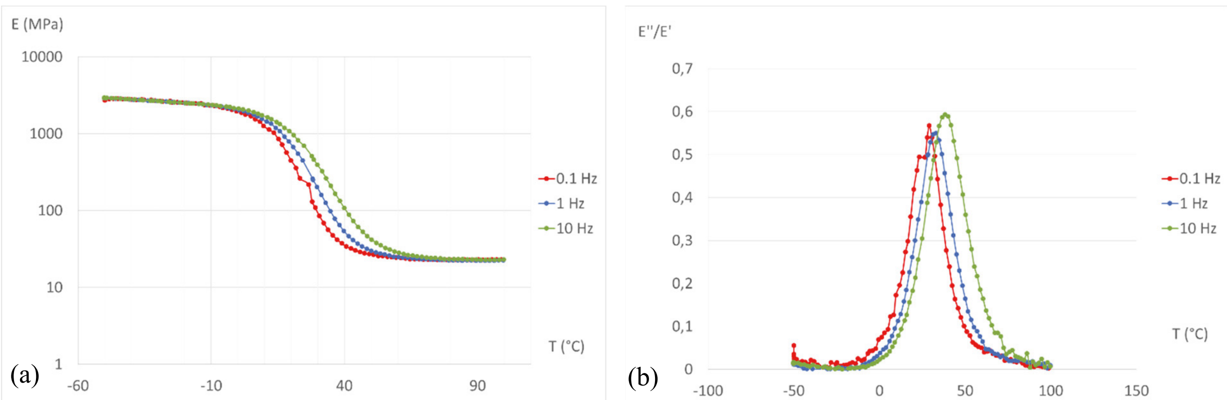

Fig. 1. DMA results for Young modulus (a) and loss factor (b)

Dynamic glass temperature transition was detected using Dynamic Mechanical Analysis with a Metravib +150 apparatus. Specimens were loaded under tensile harmonic condition using 0.1Hz, 1Hz and 10Hz frequencies and 5µm displacement amplitude ensuring linear behavior. The storage and loss modulus were measured in the [-50°C +100°C] range using 1° per minute heating rate. Then the elastic modulus $E = \sqrt{E' + E''}$ and the loss factor $tan\delta = E'/E''$ are then calculated as presented in Fig 1.

This result indicates that the SW2216 adhesive shows a 60°C large glassy to rubbery state transition, the glass temperature transition is observed at ca. 25°C which correspond to our usual operating conditions. It is then expected that our mechanical tests will be highly sensitive to temperature fluctuation, but most of all loading rate dependent.

Creep tests were also performed on specimens from the same batch. A 0.75 MPa constant stress is applied during 1 hour under 30°C ambient temperature. Six loading time conditions are tested (from 10 seconds to 400 seconds) to evaluate the influence of this parameter on the test result. Additionally, the front side of the furnace of the DMA is replaced by a window so as the specimen could be observed with a camera (Canon EOS 400D) during the test. Images are acquired every five seconds. A pattern is deposited on the specimen by spraying some white paint so that the displacement field could be detected using Digital Image Correlation (DIC) software Vic2D© (Correlate Solution). Several tests were performed on each samples. To delete previous loading and “restore” the specimen to its initial state, the specimen is heated an hour at 60°C then slowly cooled down to room temperature.

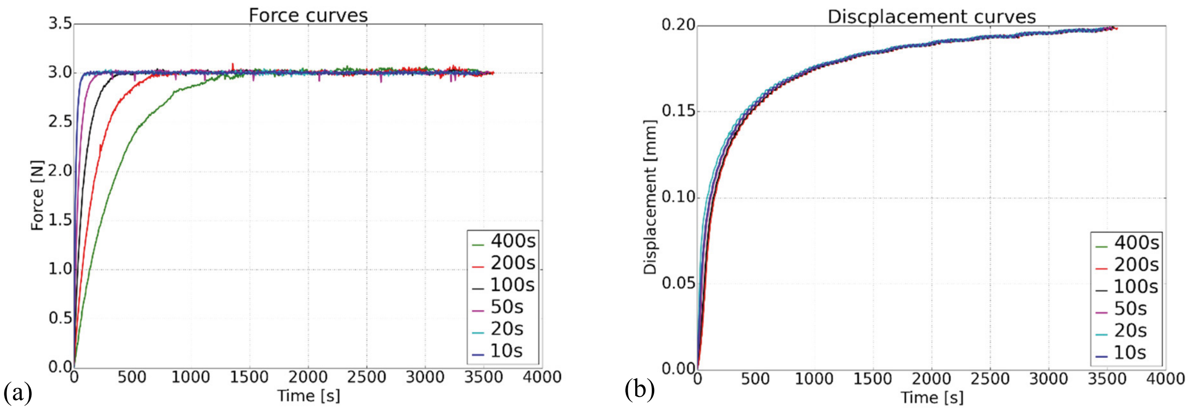

Fig. 2. Creep tests force (a) and displacement (b) curves

1.2. Double Cantilever Beam (DCB) tests

The adhesive joints mechanical properties are evaluated using crack propagation experiments in Double Cantilever Beam test arrangement. Specimens are made with two identical aluminum – zinc alloy (7075-T6) adherends. Adherend dimensions are given as follow: length: 200 mm, width: 25 mm thickness: 4.5 mm. The aluminum substrates were first sand blasted with F60 corindon particles then rinsed with acetone and dried. A silane adhesion primer was then applied and, before bonding, substrates are placed in an alignment jig to ensure correct positioning. A PTFE film is placed on both ends of the substrate surface to control the joint thickness, 200µm, as measured with an optical microscope. The same crosslinking procedure as for the bulk specimens is used here.

DCB test were performed on a Zwick tensile testing machine under a constant opening rate using three different values (0.1mm/min, 0.5 mm/min and 1mm/min). All experiments are performed at room temperature. The opening displacement Δ was measured with a 25mm range Linear Variable Differential Transformer (LVDT) sensor and the applied force is measured with a 10kN load cell.

Crack front position and deformation along the DCB specimen were observed using two digital cameras again and using DIC software again. Three strain gauges (EA-13-060LZ-120/E, Vishay micro-measurements) were bonded along the upper substrate to measure the strain along the crack propagation direction at the middle of the specimen width. The strain gauges were placed respectively at 70,120 and 170 mm from the beginning of the adherend.

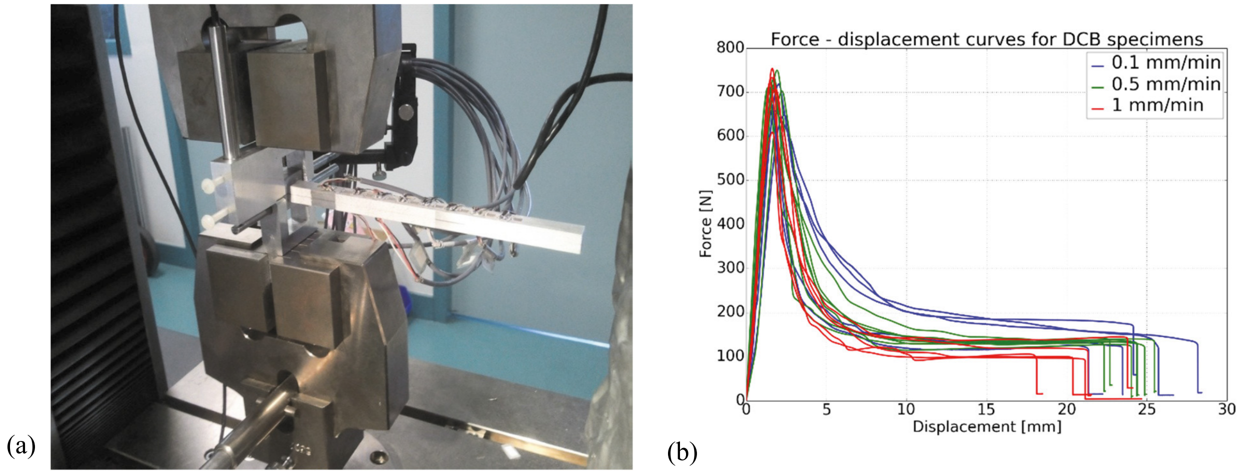

Fig. 3. DCB experimental set up (a) (Salem, 2012) and force-displacement curves (b)

2. Analysis

2.1. Creep tests

Under room temperature condition, highly viscoelastic behavior is expected. The creep tests performed on the bulk specimens are used to determine the creep compliance of the specimen but also to monitor the evolution of the apparent Poisson's ratio of the adhesive.

2.1.1. Evaluation of the adhesive stiffness

Assuming linear viscoelastic behavior, a standard linear solid model can used to describe the delayed response of the specimen. The retardation function of the material is given by the relation:

$$C(t) = C_0 + \sum_{i=1}^{N} C_i \left[1 - \exp\left(-\frac{t}{\tau_i}\right)\right] \tag{2}$$

Table 1. Average constant of the model for each loading time

Loading time (s)	C_0^{-1} (N/mm)	C_1^{-1} (N/mm)	C_2^{-1} (N/mm)	τ_1 (s)	τ_2 (s)
400	93.83	35.89	25.42	1135	129.2
200	112.2	37.84	26.32	1409	140.6
100	118.7	46.35	30.12	1029	138.6
50	184.7	58.24	42.23	514.2	65.97
20	146.2	45.79	36.13	769.7	91.99
10	192.7	47.94	42.01	838.2	96.88

With C0 being the instantaneous compliance and each constants Ci and τi control the creep behavior during a given time decade. The number N of compliances is then limited by the duration of the experiment and loading time. To take into account the non-instantaneous loading of the specimen, Boltzmann superposition principle is used to simulate the delayed deformation from the applied force history. Two time constants are sufficient to fit with reasonable precision the experimental results. The resulting compliance and time constants are summarized in table 1 for the different selected applied loading time. Under the exclusion of datas from a 50 seconds loading time, each stiffness can be fitted with a power law function and each time constant with a degree 3 polynomial law.

2.1.2. Estimation of Poisson's ratio

DIC is used to measure the transverse strain of the specimen during the creep test. Symmetric lines from the middle axis are picked. Relative displacement of those lines is calculated and its average value per image, once divided by the gap between the lines, corresponds to the transversal strain at a given time. This strain is then compared with the vertical one to estimate Poisson's ratio.

Table 2. Average Poisson's ratio for each loading time

Loading time (s)	ν
400	0.42
100	0.44
50	0.35
20	0.44
10	0.40

Apart from results for a loading time of 50 seconds, the average value is 0.42 for the adhesive Poisson's ratio.

2.2. DCB tests

The DCB test is used to evaluate the bonded joint fracture energy, Gc, during the crack propagation under mode I loading condition. Simple Beam Theory (SBT) allows simple evaluation of Gc. The adherends are modeled as simple Euler-Bernoulli beams and the bondline stiffness is supposed to be infinite. Under such assumptions the crack length, as defined by the distance between crack tip and applied load position is estimated with the relation:

$$a_{SBT} = \left(\frac{3EI\Delta}{2F}\right)^{1/3} \tag{3}$$

Δ is the opening displacement, F the applied force, E the Young's modulus of the adherend and $I = bh^3/12$ with b and h respectively the adherend thickness and width. The energy release rate is then obtained thanks to the formula:

$$G_C = \frac{12F^2 a_{SBT}^2}{Eb^2h^3} \tag{4}$$

During the stable crack propagation phase of the DCB experiment, if the adhesive exhibits a non-time-dependent behavior, GC as calculated with the relation (4) remain stable and the force decreases inversely proportional to $\sqrt{\Delta}$ (Salem et al., 2013). The observed evolution is clearly very different. A first quantification of the rate effect is obtained by calculating the indicator:

$$D = \frac{1}{n}\sum_{n}\frac{F_{SBT} - F_{exp}}{F_{SBT}} \tag{6}$$

where F_{SBT} corresponds to the theoretical F(Δ) evolution as predicted with the SBT. n is the number of experimental points in each curve describing the crack propagation. The results obtained with the three loading rate are presented in table 3. Clearly, the higher the opening rate, the more pronounced the gap between experimental results and SBT. This result is attributed to the influence of adhesive viscosity.

Table 3: Average values of the gap between SBT and experiments on the Force-displacements curves

Loading rate	Mean value of D
0.1 mm/min	0.148
0.5 mm/min	0.174
1 mm/min	0.231

2.2.1. Evaluation of crack length and crack speed

With our experimental arrangement, the crack propagation can be monitored with three different techniques. Firstly, by using the SBT formula 3, secondly with the strain gauges and thirdly thanks to image correlation. Indeed, each strain gauges signal evolution shows a peak value when the crack front crosses the strain gage position. The gauges values also allow for an estimation of the variation of the process zone size, thus enabling for the calculation of a ‘corrected’ crack length. Two camera are used to visualize the crack propagation. With the first camera, the bondline is observed at a distance 10mm to 70 mm from the applied load position, with the second one the bondline is observed at a distance 100 mm to 160mm. Using the method described by Salem et al. (2014) adherend deflection and cross section rotation are extracted from the DIC measurements. Then the image corresponding with the crack front being at the position of one of the gauge is found and the relative displacement of the adherends corresponding to crack can be estimated along with the matching critical adhesive strain. All left to be done is finding for several points of the adherend the image (i.e. time) where the relative displacement of the adherends is equals to those critical strains. These information can be compared with the asbt value to build corrected crack length evolution.

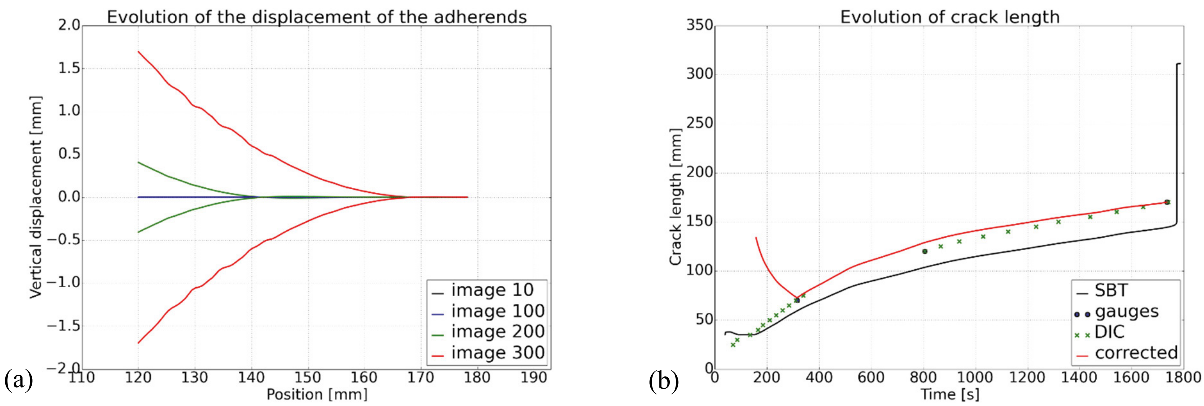

Fig. 4. Vertical displacements of the adherends (a) and comparison of estimated crack length (b) for a DCB test performed at a 1mm/ minute

Crack propagation curves are presented in figure n°4. Image correlation results (DIC) are in good agreement with both the three strain gauges indication and the method used to recalibrate values of crack length, thus proving that despite the few number of gauges it is accurate enough to be employed for future tests. Rather than the crack tip position, the crack propagation speed is a more important quantity to analyze crack propagation in viscoelastic media. Crack propagation speed is obtained by derivating asbt time evolution curve. Results show that crack speed cannot be normalized by the loading rate, so there is an influence of loading rate. In fact, the higher the loading rate, the higher the crack speed.

2.2.2. Fracture energy

Finally, the fracture energy Gc is calculated using relation (4). The resulting R-curve are presented in figure n°5. Rather than being constant, a clear decrease of the apparent fracture energy is observed during the experiment simultaneously with the decrease of the crack propagation speed. Additionally, we observe that this effect is more pronounced when the specimen opening rate is high. Obviously the fracture energy is not adequate to describe the crack propagation condition. In figure 5, the fracture energy is compared with the crack propagation rate. Again, no clear correlation is observed which outlines the need for specific fracture mechanics quantities to described the crack propagation condition along viscoelastic bonded joint.

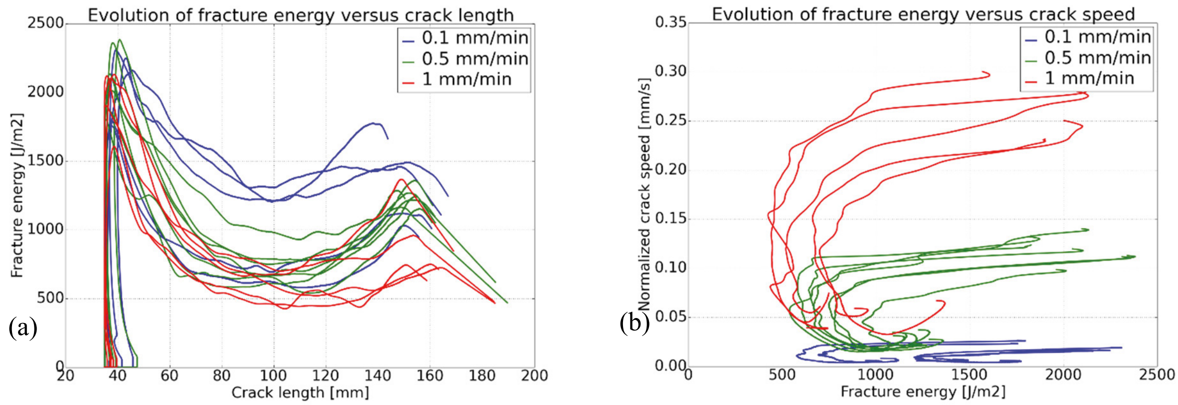

Fig. 5. Evolution of fracture energy Gc versus crack length (a) and crack speed (b)

Conclusion

Creep tests were carried out at different loading time and allowed for the evaluation of the adhesive stiffness through the calculation of its retardation function and the evaluation of its Poisson's ratio.

Three constant loading rate tests were performed on Double Cantilever Beam specimens. The results show an important gap between experimental force-displacement curves and the ones predicted by SBT. This gap was quantified for each test, allowing for an highlight of a first effect of the loading rate: the smaller the loading rate, the smaller the gap between SBT and experiments. Image correlation and strain gauge enable a reasonably precise evaluation of the crack length and the calculation of crack speed, resulting in the identification of a second effect of the loading rate: the higher the loading rate, the higher the crack speed. Calculation of the fracture energy shows that Gc does not controlled crack speed, thus invalidating its use to estimate the resistance of the joint. Future work will focus on the search of a fracture criterion involving crack propagation or crack speed.

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